THE EFFECTS OF LASER RADIATION ON PYROLYTIC SILICON NITRIDE COATINGS ON SLIP-CAST FUSED SILICA SUBSTRATES

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July 1980

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ABSTRACT

Pyrolytic silicon nitride coatings were deposited on slip-cast fused silica substrates. They were characterized and their thermal response is described. A clean heat source in the form of a continuous wave (CW) CO_2 welding laser was utilized in the experiment.

INTRODUCTION

Pyrolytic silicon nitride was deposited on slip-cast fused silica (SCFS) substrates in an attempt to form a hard coating on the softer substrates which has more desirable thermal properties than the uncoated substrate. This report will characterize these coatings and report their mechanical hardness, their spectral infrared reflectivity, and their response to continuous wave laser radiation at 10.6 μm.

EXPERIMENTAL

Pyrolytic silicon nitride coatings were deposited at 1450°C using the SiF_4/NH_3 reaction[1,2] on rectangular SCFS substrates, 2.5 cm × 1.3 cm × 5.0 cm, that had been previously fired to 1200°C and a density of 1.90 g/cm^3. After deposition, coatings and substrates were characterized using the following techniques: bulk densities of the coated substrates were determined by weighing and measuring physical dimensions; phases present in coatings and substrates were determined by X-ray diffraction analysis (XRD) on sliced sections and powders before and after laser radiation; microstructures and layer thicknesses were determined by optical microscopy; hardness of the layers was determined using a Tukon hardness tester* with a Knoop indenter and a 100-g load; surface roughness was determined on coatings and substrates with a Talysurf unit;† and reflectances were determined with a Digilab Spectrometer.‡

All laser testing was done using a continuous wave (CW) 2-kW CO_2 welding laser emitting radiation at 10.6 μm. The beam was gaussian in cross section with two major peaks. Samples were either exposed for a fixed length of time or tested to failure.

Time to failure was measured by a timer which started when the laser beam burned through a thin metal strip in front of the sample and stopped when the beam burned through a similar strip behind the sample.

RESULTS

Silicon nitride coatings were deposited on SCFS substrates at 1450°C for 1 or 2 hours. Deposits covered all six sides of the rectangular prism substrate fairly uniformly. Polished sections cut from these samples showed that the deposits had a columnar grain structure (Figure 1) in the direction of deposition and a fine uniform grain structure (Figure 2) parallel to the deposition surface. The thickness of the deposit was 16 μm. The columnar grains extended the thickness of the deposit except when growth was interrupted. Normal to the growth direction the grains had an average diameter of 9 μm, as determined using the Heyn Intercept Method.[3] Hardness values (Khn_{100g}) of 2650 kg/mm^2 were determined in the deposition plane, while Khn_{100g} equalled 3430 kg/mm^2 in the direction of growth with the

*Wilson Mechanical Instrument Co., Inc., New York, New York.
†Taylor-Hobson Ltd., Leicester, United Kingdom.
‡ Digilab, Inc., Cambridge, Massachusetts.

1. GALASSO, F. S., VELTRI, R. D., and CROFT, W. J. *Chemically Vapor Deposited Si_3N_4.* Amer. Ceram. Soc. Bull., v. 57, no. 4, 1978, p. 453-454.
2. GALASSO, F. S. *Pyrolytic Silicon Nitride Prepared from Reactant Gases.* Powder Met. Int., v. 11, no. 1, 1979, p. 7.
3. *Estimating the Average Grain Size of Metals.* ASTM Designation E 112-74, 1974 Annual Book of ASTM Standards, Part II, p. 1-33.

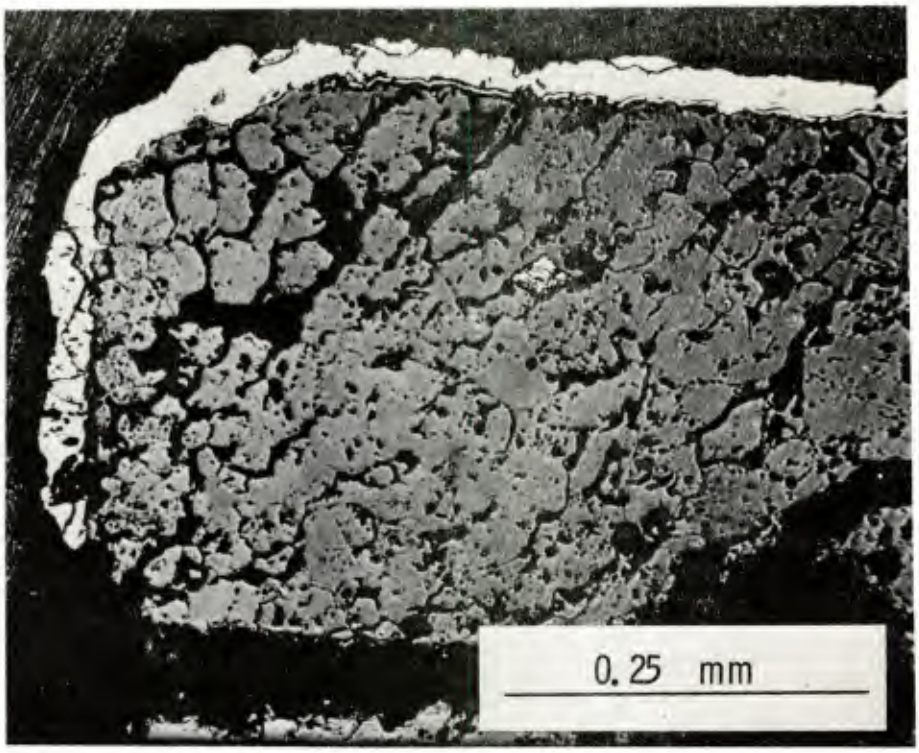

Figure 1. Photomicrograph of the corner of a slip-cast fused silica substrate (grey porous material) covered with pyrolytic silicon nitride (white material). Mag. 200X.

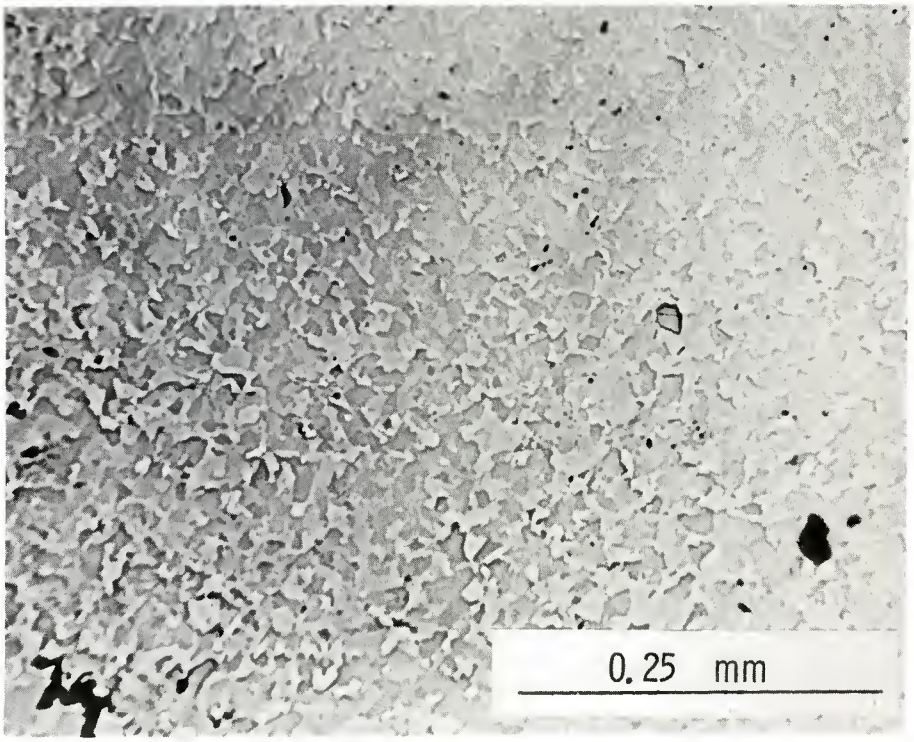

Figure 2. Polished surface of silicon nitride parallel to deposition surface showing fine grains. Mag. 200X.

indenter oriented perpendicular to the long axes of the columnar grains. When the indenter was oriented parallel to the long axis of the columnar grains, the grains cracked and delaminated, and a hardness value could not be determined. It is apparent that even thin coatings of silicon nitride increase the surface hardness of SCFS from its usual value of 560 kg/mm^2.[4]

X-ray diffraction analysis of sections cut from the samples showed that the Si_3N_4 was present in the α modification and that the SiO_2 had devitrified during the deposition process from amorphous SiO_2 to a mixture of low cristobalite and amorphous SiO_2. Further change in the SiO_2 as a result of the heating during deposition was that it increased in density from 1.90 g/cm^3 to 2.08 g/cm^3, which is a decrease in porosity from 14% to 5%. Previous work has shown that the presence of cristobalite in SCFS degrades the mechanical properties.[5] It would appear then that the increase in deposition time required to increase layer thickness would degrade the SCFS substrate.

Talysurf measurements of the deposited surfaces showed a root mean square (rms) surface finish of 99 microinches. The as-ground SCFS substrates had an rms surface finish of 40 microinches. Infrared reflectances of these materials are shown in Figure 3. Even though the silicon nitride layer has a rougher surface, it is more reflective than the as-ground SCFS.

When irradiated with a CW CO_2 welding laser at 10.6 μm, there was no discernible difference between the time to failure of the SCFS coated with silicon nitride or the uncoated SCFS, although the type of damage to the samples was different. A partial burnthrough on uncoated SCFS is seen in Figure 4. A glassy region resulting from melting is clearly visible. A damaged area on a thinly coated sample produced with a relatively low intensity beam, i.e., 320 W/cm^2, is in the form of a

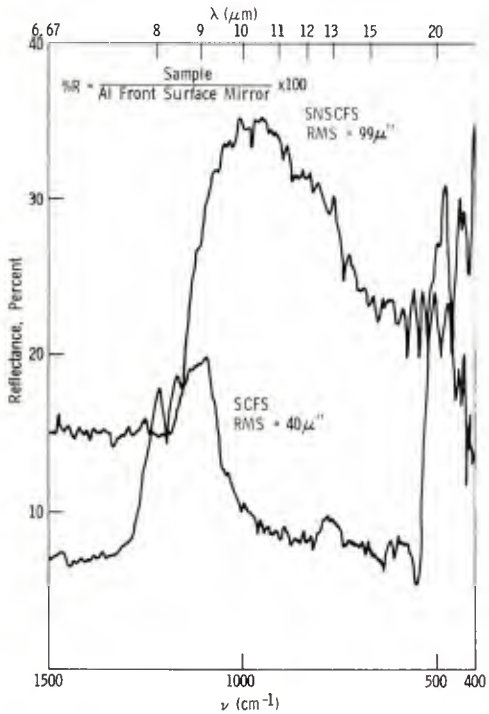

Figure 3. Infrared spectral reflectances of pyrolytic silicon nitride coatings and uncoated slip-cast fused silica expressed as a percentage of an aluminum front surface mirror. Surface finish of the two materials is given.

4. *Materials Handbook.* Corning Glass Works, Seventh Edition, August 1965.
5. HARRIS, J. N., and WELSH, E. A. *Fused Silica Design Manual: Vol I.* Georgia Institute of Technology, for the Naval Ordnance Systems Command, Contract N00017-72-C-4434, May 1973, p. 3-16.

"figure eight" because of the laser beam power profile, Figure 5. The damaged area is made up of a brownish, bubbly glass, probably SiO or an oxynitride glass, surrounded by a white powdery deposit of SiO_2. At a higher intensity of 880 W/cm^2, the centers of the layers in the "figure eight" damage areas have been vaporized away (Figure 6). A ring of metallic silicon surrounds these areas; next there is a region of crazed silicon nitride and finally a ring where the silicon nitride layer has been spalled off due to the mismatch in the coefficient of thermal expansion (CTE):

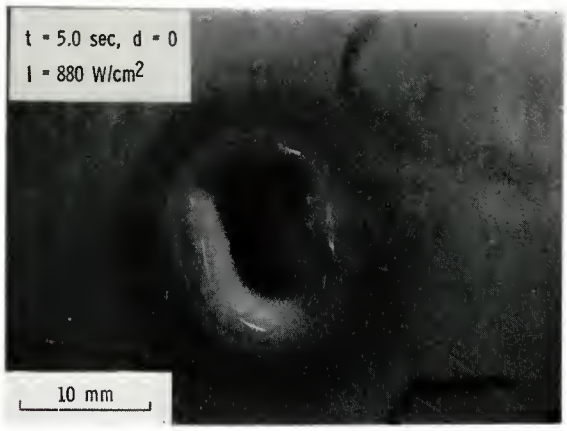

Figure 4. Damage to uncoated slip-cast fused silica after exposure to a CW 10.6-μm laser for 5 seconds at intensity I = 880 W/cm^2. Mag. 3.5X.

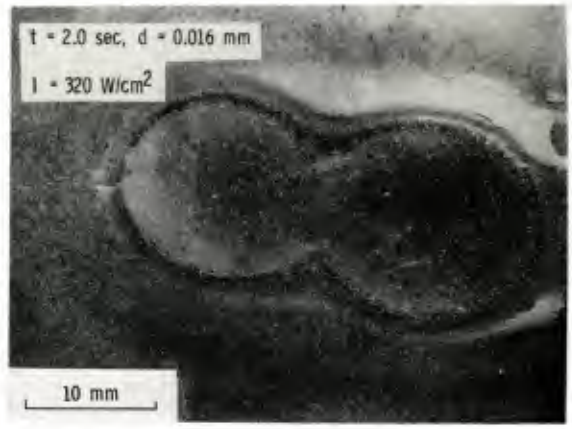

Figure 5. Damage to a 0.016-mm-thick coating of silicon nitride after exposure to a CW 10.6-μm laser for 2 seconds at intensity I = 320 W/cm^2. Mag. 3.5X.

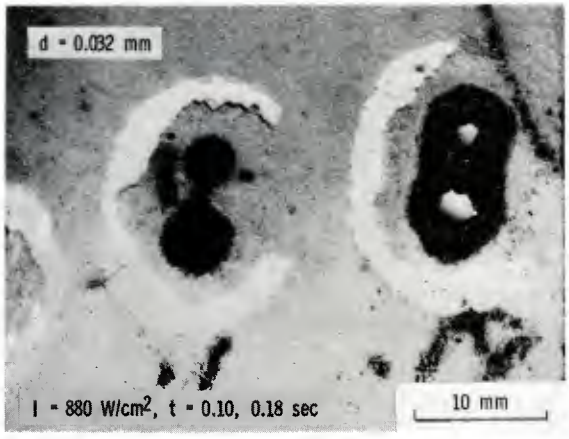

Figure 6. Damage to a 0.032-mm-thick coating of silicon nitride after exposure to a CW 10.6-μm laser for 0.10 second (left) and 0.18 second (right) at intensity I = 880 W/cm^2. Mag. 3.5X.

CTE_{SiO_2} (0-999°C) = 0.90×10^{-6} C^{-1} (Ref. 6)

and

$CTE_{Si_3N_4}$ (21-1315°C) = 3.30×10^{-6} C^{-1} (Ref. 7)

with

$\Delta CTE = 2.41 \times 10^{-6}$ C^{-1}.

At low intensities, ≈320 W/cm^2, the silicon nitride layer breaks down by an oxidation process in the reaction:

$$Si_3N_4(s) + O_2 \longrightarrow 2SiO(s) + SiO_2(s) + 2N_2\uparrow.$$

At high intensities, $\geq$ 880 W/cm^2, the reaction previously reported[8] holds:

$$Si_3N_4(s) + O_2(g) \longrightarrow 2Si(\ell) + SiO_2(\ell) + 2N_2(g)\uparrow$$

ΔH_{2100K} = -16 Kcal/mol.

Taking this reaction and the associated ΔH, we can calculate back to determine what thickness of silicon nitride layer is required to withstand a beam intensity of 880 W/cm^2 for 0.10 sec as an example.

If...

J = 88 joules/cm^2 = 0.021 Kcal/cm^2

then...

$$\frac{0.021 \text{ Kcal/cm}^2}{16 \text{ Kcal/mol}} = 0.00131 \frac{\text{mole}}{\text{cm}^2} \text{ of } Si_3N_4$$

are needed to withstand the laser beam.

With the molecular weight of Si_3N_4 = 140.28 g/mol, this is 0.184 g/cm^2 of Si_3N_4. When divided by the density of Si_3N_4, 3.2 g/cm^2, the layer thickness is found to be 0.574 mm, or 18 to 36 times greater than the thickness available in this investigation. The fact that the times to failure of the coated material were no different than that of the uncoated material was because the laser beam used for testing was overmatched with the target.

SUMMARY AND CONCLUSIONS

1. Hard silicon nitride coatings were successfully put down on slip-cast fused silica substrates, but the slow growth rates, ~16 μm/h, limited the thickness

6. LYNCH, J. F., RUDERER, C. G., and DUCKWORTH, W. H., ed. *Engineering Property Data on Selected Ceramics.* Battelle Memorial Inst., Technical Report AFML-TR-66-52, June 1966, p. 5.4.6-10.
7. *Engineering Property Data on Selected Ceramics, Vol. I, Nitrides.* MCIC Report MCIC-HB-07, Volume I, March 1976, p. 5.3.3-2.
8. VIECHNICKI, D. J., MEYER, F. P., and PETSCHKE, C. *Response of Fused Silica and Silicon Nitride to HEL Irradiation.* Army Materials and Mechanics Research Center, AMMRC TR 78-31, July 1978.

of the coatings, and the elevated temperatures, 1450°C, devitrified the previously X-ray amorphous substrates with a possible degradation in mechanical properties.

2. There was no effect of the silicon nitride coatings on overall laser response because the layers were too thin and were overmatched by the laser beam.

3. At the relatively low intensities of 320 W/cm^2, the laser beam heated the silicon nitride layer and caused it to oxidize to a glass. At intensities of 880 W/cm^2 and greater, the laser beam heated the silicon nitride more rapidly and caused the silicon nitride to decompose to silicon and silica. Crazing and spalling of the layer occurred at a small distance from the area the beam impinged on, with more pronounced spalling with increased layer thickness. This is due to the mismatch in thermal expansion coefficients between silicon nitride and silica. This also implies that thicker silicon nitride layers (thick enough to significantly change the response of the material) would be more apt to spall off during subsequent laser radiation.

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